Microprobe Attachment for Quantitative Studies of Cathodo-Luminescence

by

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A versatile monochromator-photomultiplier attachment to an electron microprobe has been developed. It has facilitated the study of a number of cathodo-luminescence phenomena including: 1) recording of luminescence spectra, 2) distribution of luminescent phases in specimens, 3) growth and decay of "F-center"-induced luminescence and 4) relation of luminescent intensity to composition within a phase. This paper describes the salient features of the attachment and illustrates its application to several pertinent problems.

The attachment consists of a monochromator housing, a monochromator, an ocular tube, a selection of interchangeable slits and interchangeable photomultiplier detectors. The interchangeable components are readily permuted so that one can perform a variety of experiments or use the microscope in the normal manner without having to remove the attachment. For visual microscopic examination, the ocular is inserted in the attachment. Then the specimen can be viewed either through the monochromator, in which case the specimen is displayed in any color light desired, or alternatively, the monochromator can be bypassed for normal microscopic observation. Defining slits for the monochromator are inserted in place of the ocular, and then photomultiplier tubes in light-tight housings are slipped onto the attachment for electronic detection of light intensities.

The attachment replaces the ocular tube of the optical microscope on the microprobe. The monochromator within the attachment is an inexpensive Bausch and Lomb wedge interference filter of narrow band width and high transmission (average linear dispersion of 5.5 millimicrons per mm) with a useful wavelength range of 400 to 700 mu. An odometer is used to read wavelength directly to the nearest 0.1 mu. The visible spectrum is scanned by use of a synchronous motor drive on the monochromator with strip chart recording of the signal. A complete spectrum of a luminescent area, within the contour of interest can be obtained. The amplified photomultiplier signal can also be displayed on the oscilloscope in a fashion analogous to the conventional electron backscatter image as described earlier by Heinrich (1). However, with this attachment light intensity displays at a particular wavelength can be photographically recorded.

Measurements of the luminescence of individual enstatite grains from a suite of enstatite chondrite and achondrite meteorites, produced by moderate-energy electron excitation (less than 40 keV), have established spectral energy distributions and excitation efficiencies as a function of mineral composition, irradiation time, and electron energy within individual grains. Striking variations of composition and luminescence spectra within the individual grains have been found, indicating the importance of precise localized investigation of a phase assemblage to provide an understanding of the bulk luminescent response of the material. The influence of increasing manganese

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activator ion concentration and specimen purity is demonstrated with a shift of wavelength to longer wavelengths and higher intensity, and the quenching influence of iron is confirmed. The signals from these weakly luminescent silicates are adequate and do not require photomultiplier-differential current amplifier circuits or photomultiplier unit cooling for their detection and quantitative measurement.

In many instances, the cathodo-luminescent pattern has been used as a "marker" to establish the original reaction interface in solid state reactions of oxide materials.

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Reference

1. K. F. J. Heinrich, "Oscilloscope Readout of Electron Microprobe Data," Advances in X-ray Analysis, Vol. 3, University of Denver, Plenum Press, New York, 1962, pp. 291-300.